EXTRACTION OF PATCHOULI OIL USING STEAM DISTILLATION

DEWI HARYANI BINTI SULAIMAN

A thesis submitted in fulfillment of the
requirement for the award of the degree of
Bachelor Degree of Chemical Engineering

Faculty of Chemical and Natural Resources Engineering
Universiti Malaysia Pahang

MAY 2008

DECLARATION

I declare that this thesis entitled "*Extraction of Patchouli Oil Using Steam Distillation*" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature	:	……..
Name	:	Dewi Haryani binti Sulaiman
Date	:	14 May 2008

To my beloved parents, siblings, and fiancé

ACKNOWLEDGEMENT

I wish to express my deepest gratitude to my supervisor, Miss. Suriyati binti Saleh for her tireless efforts and on-going support, advice as well as guidance, without whose help, my thesis would not have been completed successfully. I am also very thankful to my panels who have given useful suggestion and comments.

I would like to extend my heartfelt gratitude to all my friends who offer tips, advice and endless cooperation especially to postgraduate students who help to contribute ideas to my thesis.

My appreciation to beloved parents, papa and mummy, for giving me support in all aspect like money and care to complete this thesis. Thank you to my siblings for their undivided courage support and also to my fiancé Syed Abdullah bin Syed Ahmad, who is always beside me for his moral support and love. I love you all.

Last but not least, to others who have contributed direct or indirectly to the writing of this thesis. Your views and tips are very useful and priceless indeed. Thank you.

ABSTRACT

This study is about Extraction of Patchouli Oil Using Steam Distillation. The objective of this research is to study the feasibility of the optimum condition of steam distillation in patchouli oil extraction process. Patchouli oil extraction is still new but has gained large market demand for the benefit on therapeutic and healing properties of this essential oil. However, cost-effective route have yet to be develop. This research has identified two scope of study to achieve the objective which is to vary the effect of different extraction time and sample mass on the yields. In this extraction, part of the plant used is the leaves and stick. Firstly, the raw material is exposed 3 hours under direct sunlight and 3 days in room temperature. Dried patchouli plants are then cut to 2 cm in size. Then, the leaves are stacked in the extraction vessel. High pressured steam passed through the plant material from the bottom of the vessel. Hot steam will force open the pocket in which the essential oil of the patchouli was kept. Next, the steam which contains the essential oil passed through cooling system to condense the steam which would separate the essential oil from water. Pure oil is extracted with this method. For this equipment with the range of 7 hours extraction time and 2 kg to 4 kg sample masses, the optimum extraction time is at 7 hours with 3 kg sample mass.

ABSTRAK

Projek ini bertajuk Pengekstrakan Minyak Nilam Menggunakan Penyulingan Berwap. Objektif projek ini ialah untuk mengetahui keadaan yang paling optimum untuk penyulingan berwap dalam melakukan proses pengekstrakan ini. Pengektrakan minyak nilam adalah masih baru di pasaran tetapi permintaan terhadap kebaikan minyak ini dari segi perubatan terapi telah mendapat sambutan meluas. Namun begitu, proses yang efektif dari segi kos masih lagi dalam kajian. Dalam projek ini, dua parameter telah ditetapkan untuk mencapai objektif projek ini iaitu kesan perubahan masa pengekstrakan dan berat nilam terhadap hasil penyulingan. Dalam proses ini bahagian yang digunakan ialah daun dan batang pokok nilam. Pertamanya, nilam perlu dikeringkan di bawah sinaran matahari selama 3 jam dan suhu bilik selama 3 hari. Nilam yang sudah dikeringkan itu di potong dalam anggaran saiz 2 cm. Kemudiannya, nilam itu akan dimasukkan ke dalam ‘vessel’ penyulingan dan wap air bertekanan tinggi akan di alirkan dari bawah ‘vessel’. Wap air yang panas ini akan menyebabkan minyak yang berada di dalam poket tumbuhan akan terbuka. Campuran wap air dan minyak nilam akan terus mengalir ke sistem penyejukan dan terkondensasi di mana air dan minyak nilam ini akan terpisah. Minyak yang asli kebayakannya mengunakan cara ini bagi proses pengeksrakan. Bagi alat ini untuk julat masa 7 jam masa pengekstrakan dan bagi berat jisim sampel di antara 2 kg hingga 4 kg, keadaan optimumnya ialah pada 7 jam masa pengekstrakan dan pada 3 kg berat sampel.

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LIST OF SYMBOLS

USD $	-	United State Dollar
kg	-	Kilograms
RT	-	Retention Time
%	-	Percentage
>	-	more than
±	-	Estimation (plus minus)
^{o}C	-	Degree celcius
T	-	Temperature
cm	-	Centimeter
atm	-	Atmospheric pressure

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Patchouli is an essential oil obtained from dried leaves or of a plant of the same name. Its botanical name is *Pogostemon Cablin*. Patchouli oil is widely used in perfumes as one of the important natural essential oils used to give a base, lasting character and fixative ability to a fragrance. It is originated from East and West Indies and their name derives from the Tamil *patchai* (green), *ellai* (leaf). Patchouli is also known as patchouly, *tamala pattra* in Sanskrit and *guang huo xiang* in Chinese. Indonesia is the major producer of patchouli oil in the world with and estimated 550 tons per year, which is more than 80% of the total (Robbins, 1983; Tao, 1983). The taxonomic position of patchouli is given below in Table 1.1 (Wikipedia, 2007. *Patchouli*).

Table 1.1: Taxonomic position of patchouli

TAXONOMIC POSITION	
Kingdom	Plantae
Division	Magnoliophyta
Class	Magnoliopsida
Order	Lamiales
Family	Lamiaceae (Labiatae)
Genus	Pogostemon
Species	P.cablin

Before it became popular in Europe, the unique patchouli odor was being impregnate in the Indian shawls and Indian ink. Despite being used as alternative lifestyle in modern industry in fine products like cosmetic product as well as a component in about a third of modern, high-end perfumes, including more than half of perfumes for men, patchouli is also an important ingredient in East Asian incense. It is also used in paper towels, laundry detergents, and air fresheners as a scent in products.

Patchouli is a very fragrant, bushy herb with soft oval leaves and square stems. It grows from two to three feet in height and provides an unusual odor that is nonetheless characteristic of patchouli when the leaves are rubbed. The plant grows well in southern climates. It enjoys hot weather but not direct sunlight. If the plant withers due to lack of watering it will recover well and quickly once it has been watered. The seed-bearing flowers are very fragrant and bloom in late fall. The tiny seeds may be harvested for planting, but they are very delicate and easily crushed. Cuttings from the mother plant can also be rooted in water to produce further plants. Patchouli is a tropical member of the mint family. Leaves are harvested several times a year, dried, and exported for distillation of the oil, although the highest quality oil is usually produce from fresh leaves, distilled close to the plantation (Wikipedia, 2007. *Patchouli*).

An important component in a patchouli oils is patchouli alcohol ($C_{15}H_{26}O$) or patchoulol known as terpene. Generally, patchouli oil consists of over 24 different sesquiterpen es. One of the organic compounds responsible for the typical patchouli scent is the optical isomer. All the chemical compositions in the patchouli essential oil are analyzed by using the Gas Chromatography Mass Spectrometry technology.

1.2 Problem Statement

At present, the essential oil industry is not only focusing to the production and distribution of essential oils alone, but has focus more on the improvement of methods

and maintenance of standard quality. This is because of the large market demand for the benefit in therapeutic and healing properties of the essential oils. Patchouli oil is an important ingredient in many fine fragrance products but the patchouli oil extraction is still new compared to other essential oil extraction. However, cost-effective route to produce the oil has yet to be developed. Furthermore, the price of patchouli oil increasing by years (Lerner and Ivan, 2003).This study focuses specifically on extraction of patchouli oil, as it is widely appreciated for its pleasant characteristic and long lasting odor, and to find the best method for extraction. The extraction method used in this study is steam distillation as patchouli is commonly extracted using this method.

1.3 Objective of the Study

The objective of this research is to study the feasibility of the optimum condition of steam distillation in patchouli oil extraction process.

1.4 Scope of Study

In order to achieve the objective, the following scopes have been identified:

1. Effect of different extraction time on oil yield
2. Effect of different sample mass on oil yield.

For the first parameter, extraction time, the sample mass is fixed for 6 hours duration where the oil yield will be record for every one hour starting from 3 hours. The same experiment will be run for three times to get the average reading for each extraction time. For the second parameter the yield will be recorded at different sample mass ranging from 2 kg to 4 kg and the yield will be record at certain period. The experiment also will be run for 7 hours.

CHAPTER 2

LITERATURE REVIEW

2.1 Essential Oil

Early in history, the term “essential oil” or ”ethereal oil” defined as the volatile oil obtained by the steam distillation of plants. Essential oils which are also referred to as “essences”, not only originated from flowers, but from herbs, shrubs, trees and various other plant materials.

Gradually with the advance knowledge in science arise improvement in the methods of preparing the oils, parallel with the development of a better knowledge understanding of the constituent of the oils. It was found that the oil contains chiefly liquid and more or less volatile compound of many classes of organic substances usually dependent upon the oxygenated compounds. Four main groups, which are characteristic of the majority of the essential oils, i.e.:

1. Terpenes, related to isoprene or isopentene;
2. Straight-chain compounds, not containing any side branches;
3. Benzene derivatives;
4. Miscellaneous

(Haagen-Smit, 1949)

Large volumes of oils are usually distilled from leafy material such as lemongrass, citronella and cinnamon leaves. Meanwhile, the small volumes of oils are

usually distilled from fruits, seed, buds and flowers (e.g cloves, nutmeg and coriander). The percentages of characteristic differ at different part of the plant.

2.1.1 Benefit of Essential Oil

The plant derives specific benefit from its own essential oils. The two major advantages they gain from their essential oil are as protection and reserve food. The irritating effect of many oils affords a degree of protection against the depredations of animals and plant parasites. In individual cases a contribution is made toward more effective pollination through insect visits. The action of some essential oils is similar in certain respect to that of anaesthetics on animal cells. The inhibiting and damaging effect of the oils on many life processes has been turned to our advantage in the use of these compounds as bactericidal and fungicidal agents. In other cases of protection, plants which emanate a considerable amount of oils are prevented from becoming too warm since heat is absorbed in the vaporization of the oils. In this way the oil function as a water-sparing mechanism.

As for human being, the essential oils were regularly used in ancient Rome, Greece, and Egypt and throughout the Middle and Far East. Their common feature, the essence of a plant; an identifiable aroma, flavour, or other characteristic was of some practical use. They were used as perfumes, food flavours, deodorants, pharmaceuticals, and embalming antiseptics. For example, the essential oil is the primary ingredient in aromatherapy treatments which are safe and simple natural product.

2.1.2 Physical Properties

Most plants contain essential oils but only the aromatic plants produce essential oil in sufficient quantities. They can be more or less fluid of which some are viscous; others are fairly solid and most are watery. They are sometimes resinous and often have

a coloring, which ranges from yellow to emerald green and blue to dark brownish red. Essential oils are diffuse and penetrating. Their high degree of vibratory capacity gives them this quality. Therefore, they should be kept in well stopper with a sound cork. Other than that, as essential oils are very sensitive to heat and light, they should be stored in a cool place and dark bottles. In order to minimize oxygen exposure, a small amount of oil in a large bottle should be transferred to a small bottle instead. Another important physiochemical criterion of the quality and purity of an essential oil is the specific gravity which the values vary between the limits of 0.696 and 1.188 at 15°C which in general, the gravity is less than 1.000 (Gildemeister *et al.,*1956). Essential oil at 15°C/15°C of specific gravity defined as the ratio of the weight of a given volume of oil at 15°C to the weight of an equal volume water at 15°C. Essential oil boil generally between 150^{o}C-300^{o}C (consist of many compound) however the compound are steam volatile and can be distillate at around at 100^{o}C.

2.1.3 Chemical Properties

Essential oil chemical properties are usually analyze with a chromatography and the primary components are terpene hydrocarbons (monoterpene hydrocarbons and sesquiterpenes), oxygenated compounds consists of phenols and alcohols (monoterpene alcohols and sesquiterpene alcohols), aldehydes, ketones, esters, lactones, ethers and oxides.

Most essential oils consist of mixtures of hydrocarbons (terpenes, sesquiterpenes, etc.), oxygenated compounds (alcohols, esters, ethers, aldehydes, ketones, lactones, phenols, phenol ethers, etc.), and a small percentage of viscid or solid nonvolatile residuen (paraffin, waxes, etc.). Of these the oxygenated compound are the principal odor carriers, although the terpenes and sesquiterpenes, too, contribute in some degree to the total odor and flavor value of the oil. The oxygenated substances posses the added advantage of better solubility in dilute alcohol and, with the exception of some aldehydes, of greater stability against oxidizing and resinifying influences (Guenther, 1949).

Essential oils that are rich in monoterpenoid constituent were those obtained from the leaves and fruit peels of *C. hystrix*, the fruit peel of *C. aurantifolia,* and the leaves of *O. citriodorum*. Limonene, α-pinene, β-pinene, linalool, geraniol, citral, terpinen-4-ol and alpha-terpineol were the major representatives of monoterpenoids present. Essential oils that are rich with sesquiterpene constituent and phenyl propanoids were *O. tenuiflorum* and *P.cablin* (Nor Azah *et al*., 2007).

The first primary component found is the terpenes hydrocarbons. One of it is the monoterpene compounds that are found in nearly all essential oils and have a structure of 10 carbon atoms and at least one double bond. The other one is the sesquiterpenes consists of 15 carbon atoms and has complex pharmacological actions.

Another primary component is the oxygenated compound consists of phenols and alcohols. Phenols found normally have a carbon side chain. Due to the nature of phenols, essential oils that are high in them should be used in low concentrations and for short periods of time, since they can lead to toxicity if used over long periods of time, as the liver will be required to work harder to excrete them. It can also cause skin and mucus membrane irritants and although they have great antiseptic qualities, like cinnamon and clove oil, they can cause severe skin reactions. Alcohols found in the essential oil like monoterpene alcohol on the other hand have good antiseptic, anti-viral and anti-fungal properties with very few side effects such as skin irritation or toxicity and have an uplifting energizing effect. As for sesquiterpene alcohols, they are not commonly found in essential oils but when found they have great properties, which include liver and glandular stimulant, anti-allergen and anti-inflammatory. For the rest of the other components, aldehydes, ketones, esters, ethers, and oxides are found in a small quantity.

2.1.4 Types of Essential Oil

Today, there is a lot of essential oil from different kind of aromatic plant. Table 2.1 show some of the plants plant used in the extraction of essential oils.

Table 2.1: Types of essential oil

Plant	Part of Plant Used	Botanical Name	Country of Origin	Important Constituent	Properties
Chamomile	Flower	*Matricaria recutita*	England, Germany, France, Morocco	Bisabolol	Sedating, nurturing, soothing, calming, reassuring
Cinnamon	Leaf	*Cinnamomum zeylanicum*	Sri Lanka, India	Eugenol	Condiment and flavouring material, anti-oxidant, antimicrobial
Lavender	Flower	*Lavendula intermedia*	England, France, Yugoslavia, Bulgaria	Linalol	Anti-depressant, appeasing, balancing, , purifying, relaxing, sedative, soothing
Lemongrass	Leaf	*Cymbopogon spp*	Tanzania	Citral, Citronella, Terpenes	Analgesic, anti-depressant, antimicrobial. antipyretic, antiseptic, astringent, bactericidal
Sandalwood	Wood	*Santalum Album*	Nepal, Sri Lanka, Hawaii	Santalol	Antimicrobial, antiseptics
Clove	Bud	*Eugenia Caryophyllus*	Indonesia	Eugenol	Carminative, anthelmintic, anodyne
Turpentine	Resin	*Pinus spp*	Mediterranean country	Terpenes	Solvent for paints, antiseptic, Diuretic

2.1.5 Effect of Extraction Process to Essential Oil

A number of factors determine the final quality of a steam distilled essential oil. Aside from the plant material itself, most important are time, temperature and pressure, and the quality of the distillation equipment. Essential oils are very complex products; each is made up of many, sometimes hundreds, of distinct molecules which come together to form the oil's aroma and therapeutic properties. Some of these molecules are fairly delicate structures which can be altered or destroyed by adverse environmental conditions. So, much like a fine meal is more flavorful when made with patience, most oils benefit from a long, slow 'cooking' process. The temperature of the extraction chamber cannot be too high, lest some components of the oil be altered or destroyed.

The same is true of the chamber's pressure. Lavender essential oil, for example, should not be processed at over 245^{o}F and pressure at 3 psi. Higher temperatures or pressures result in a 'harsh' aroma, more chemical than floral and lessen the oil's therapeutic effects. Also, the extraction period must be allowed to continue for a certain period of time in order to flush all the oil's components from the plant, as some are released more quickly than others.

Despite the drawbacks of aggressive processing, high temperatures and pressures are often used to produces large quantities of oil in a short period of time. These oils are usually destined for use in cosmetic and processed food manufacturing, but are sometimes sold to final consumers as essential oils for use in aromatherapy. These oils will be less expensive, but are of limited therapeutic value, and the difference is apparent when the aromas are compared side-by-side. (The Ananda Apothecary, 2007. *Making Essential Oil-Steam Distillation CO_2 and Absolutes*)

2.2 Patchouli

2.2.1 Introduction

Patchouli or scientifically known as *Pogestoman cablin (P.cabin)* is a member of the mint family. Patchouli is perennial, bushy plant that grows up to three feet high, with a sturdy, hairy stem and large, fragrant, furry leaves, about four inches long and five inches across as shown in Figure 2.1 (Aroma-pure,2007.*Patchouli*). It has whitish flowers tinged with purple as shown in Figure 2.2 (Anniesremedy,2007.*Herb*). It grows in tropical climates. The plant originated from India and Indonesia. However become popular in the west beginning around 1844 when the first dried leaves, Figure 2.3 (Scents-of-earth,2007.*Patchouli*), arrived in London. Before that, it was a well-known fragrance in Indian textiles throughout Europe. It is used as an insect repellant and perfume. It is a base note in several famous perfume ingredients for both men and women. It was grown in China almost two thousand year ago and was used as a perfume for ink. Today, it is commonly used in cigarettes to compensate for a lack of taste due to reduced tar content. Good quality patchouli will retain its sweeter notes on a perfume blotter for months. The leaves must be fermented, Figure 2.4 (Alchemy-works,2007.*Herb*), during the process before they could produce the full blast of their scent.

Figure 2.1: Bushy of patchouli leaves